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14. ABSTRACT The study was to determine if the microstructure of melt processed Ag-sheathed $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ (2212) tape could be improved by crystallizing 2212 from its primary phase field. Ag-sheathed tape was fabricated with BSCCO powder whose composition was in the Bi-2212 primary field. It was melt processed. It was learned when analyzing the melt processed tape that the powder that was synthesized was contaminated with Teflon. It had decomposed and reacted with the BSCCO forming SrF_2 that shifted the overall BSCCO composition out of the primary phase field. There was not time to synthesize uncontaminated powder in the study. Instead, the effect of the heating rate through the melting point of 2212 was studied. It was found that fully processed tape heated through the melting point at $1^\circ\text{C}/\text{min}$ had the most homogeneous microstructure. Faster rates led to porosity in the core or ballooning of the Ag-sheath, while slower rates led to large, remnant nonsuperconducting phases in the fully processed tape.					
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Final report
Optimization of J_c in BSCCO Tapes
Grant # N00014-99-1-0323

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Summary

This research was to investigate increasing J_c in 2212 conductors by improving the microstructure (i.e., eliminating nonsuperconducting phases) by forming 2212 by melt processing from the 2212 primary phase field. In the primary phase field, the 2212 phase would form directly from the melt that is free of crystalline nonsuperconducting phases. In the actual study, the powder we synthesized was contaminated with F from the Teflon filter we used in the synthesis. We switched to Teflon filters because the silver filter we previously used to make the powder was no longer manufactured. The F present in the melt was tied up as SrF_2 , which changed the overall composition of the liquid. Because this project only had funding for six months of graduate student time, there was not enough time to synthesize new powder. Instead we switched to study the effect of heating rates through the melting point of 2212 on the microstructure of the melt at T_{max} and fully processed tape. We found three regimes for the heating rate: slow heating ($0.17^\circ\text{C}/\text{min}$) led to large nonsuperconducting second phases in the melt; fast heating ($5^\circ\text{C}/\text{min}$) led to bubbles in the melt; intermediate heating ($1^\circ\text{C}/\text{min}$) gave moderate sized second phases and no bubbles in the melt. Details are provided below.

Primary phase field studies

We synthesized powder with the composition $\text{Bi}:\text{Sr}:\text{Ca}:\text{Cu} = 1.88:1.73:1.23:2.16$, which Winnie Wong-Ng and Larry Cook at NIST had calculated was in the 2212 primary phase field. We used aerosol spray pyrolysis, which is our standard method for making BSCCO powder. Prior to this synthesis in this study, we separated the powder from the flowing gas stream using a silver filter, which was a thin sheet of silver that contained micron sized holes. The manufacturer had quit manufacturing the large size silver filters, so we decided to switch to a Teflon filter. After we made the powder, fabricated Ag-sheathed 2212 wires from the powder, and did the heat treatment studies, we found SrF_2 in the BSCCO core. We determined this was due to Teflon that had come off the filter and contaminated the BSCCO powder. The Teflon reacted with the BSCCO powder forming SrF_2 during the powder processing steps in which the nitrates and carbon were removed from the aerosol spray pyrolysis powder.

Our quench studies with the F contaminated powder showed the melt contained crystalline nonsuperconducting phases in the melt. Figure 1 shows the microstructure of a sample quenched from 881°C . One of the phases in the melt is SrF_2 . The presence of

F, which was tied up as unreactive SrF_2 , shifted the overall BSCCO composition, most probably out of the primary phase field. Due to this project only providing funding for six months of graduate student time, there was not time to synthesize F-free powder. Instead we studied heating rates through the melting point of 2212.

Studies of the heating rate through the melting point of 2212

We studied the effect of the heating rate through the melting point of 2212. When 2212 melts, it releases O_2 , which must diffuse through the Ag sheath in Ag-sheathed tapes. If the heating rate is too high, there isn't sufficient time for the O_2 to diffuse through the Ag and the tape can bubble. At very fast rates, these bubbles cause the tape to balloon out, destroying the tape geometry. At slower rates, bubbles can form that are not apparent looking at the outside of the tape, but are seen as internal bubbles (porosity) in the microstructure of the core. We used our normal Ag-sheathed 2212 tape with the overall BSCCO composition of 2.1:2:1:1.95 for the study.

Figure 2 shows the heating rates we used through the melting point of 2212. 2212 begins to melt at 865-870°C. Figure 3 shows the microstructure of fully processed tape processed with different heating rates. We were initially surprised at the microstructure of the slowly heated tape that contained many large remnants of the crystalline nonsuperconducting phases that were present in the melt. However, additional quench studies from T_{max} of slowly heated samples showed that these nonsuperconducting phases had already grown large in size during the slow heating. They were large in the fully processed tape because they had not fully reacted during cooling when the 2212 phase formed.

We found that fully-processed tape that had been heated at 1°C/min had the best microstructure. Fortuitously, this was the heating rate that we had been using prior to this study, so we have continued to heat through the melting point at 1°C/min.

Publications and Presentations:

No publications came out of this research. The results were presented at Deborah Van Vechten's program review in Cape Cod, MA on March 5-7, 2000.

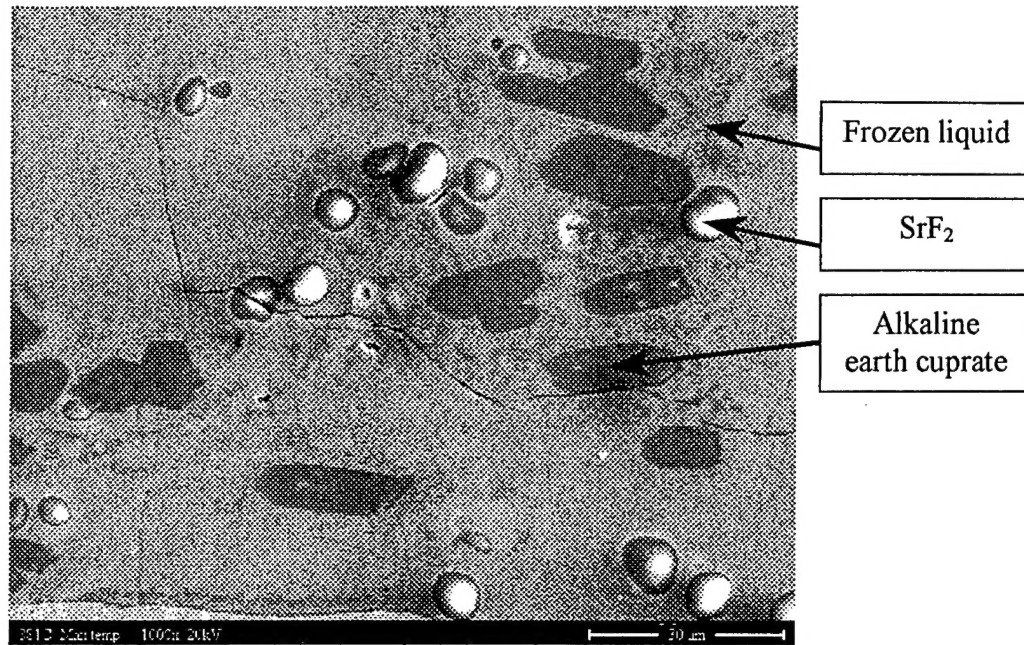


Figure 1. Cross section of the BSCCO core in a Ag-sheathed tape that was quenched from 881°C. The BSCCO powder was initially contaminated with Teflon that decomposed forming SrF_2 . The phases present in the core are frozen liquid, alkaline earth cuprate, and SrF_2 .

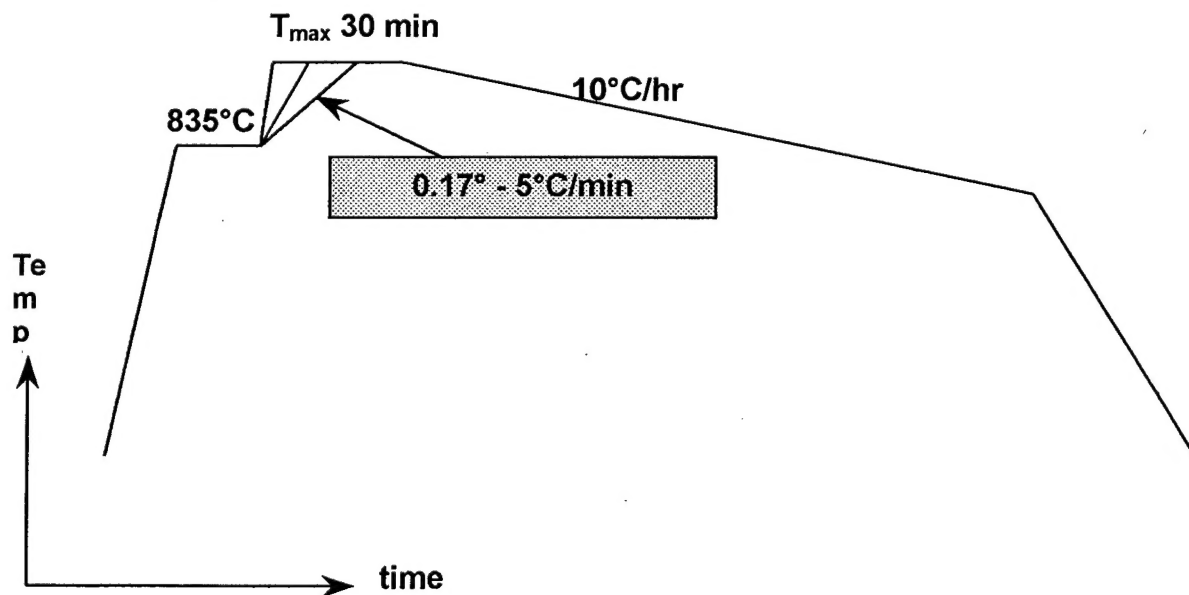
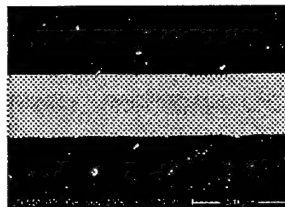


Figure 2. Processing schedule to study the effect of heating through the melting point of 2212 at different rates. 2212 begins to melt between 865-870°C. $T_{\text{max}} = 890^\circ\text{C}$.

0.17°C/min



1.0°C/min



5.0°C/min

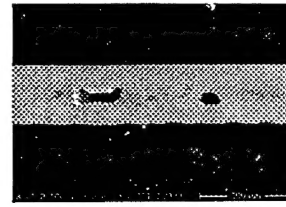


Figure 4: Longitudinal cross sections of fully processed Ag-sheathed 2212 tape with different heating rates. Note the large, remnant, nonsuperconducting phases present in the tape processed with 0.17°C/min heating. The thickness of the this tape had also decreased. The best microstructure, and presumably highest J_c , was obtained using 1°C/min. At 5°C/min, the fully processed tape contained large pores that had formed when O_2 evolved during melting. The heating rate was slow enough that the O_2 that evolved did not cause the Ag sheath to balloon, i.e., the dimensions of the tape had not increased significantly during melting due to O_2 evolving during melting.